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(54) Title: CALCIUM CARBONATE COMPOSITIONS FOR USE IN THE MANUFACTURE OF TOOTHPASTE

(57) Abstract: An aqueous suspension of particulate calcium carbonate for use in the manufacture of toothpaste compositions includes in addition to the calcium carbonate one or more dispersing agents and one or more preservative agents and wherein the aqueous medium of the suspension has been treated so that the biological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA). The calcium carbonate may comprise a precipitated and/or ground calcium carbonate. The suspension may have a solids content of from 50 % to 99.5 % by weight. The suspension may be flowable and pumpable.

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(54) Title: CALCIUM CARBONATE COMPOSITIONS

(57) Abstract: An aqueous suspension of particulate calcium carbonate for use in the manufacture of toothpaste compositions includes in addition to the calcium carbonate one or more dispersing agents and one or more preservative agents and wherein the aqueous medium of the suspension has been treated so that the biological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA). The calcium carbonate may comprise a precipitated and/or ground calcium carbonate. The suspension may have a solids content of from 50 % to 99.5 % by weight. The suspension may be flowable and pumpable.

CALCIUM CARBONATE COMPOSITIONS

The present invention relates to calcium carbonate compositions, especially for use in the manufacture of toothpaste compositions.

Utilized as a polishing agent in toothpaste formulations, calcium carbonate, especially precipitated calcium carbonate (PCC) a chemically synthesized form of this product, is presently supplied to toothpaste industries as a dry powder. Using this product in this form causes a series of problems, such as: a) insalubrious conditions at the production site, owing to the presence of large amounts of powder; b) the need to use paper bags to contain and store the powder; c) low productivity rates, owing to the time spent in the addition of powdered carbonate to other ingredients in the toothpaste manufacturing process; d) the need to keep a large storage of the product close to the toothpaste manufacturing plant; and e) extensive handling of the product during the processing stages, increasing the risk of causing microbiological contamination in the final product.

Calcium carbonate is already supplied to other industries, e.g. the paint and paper industries, in aqueous slurry form. However, the slurries available would not be suitable for use in the toothpaste industry. The purpose of the present invention is to provide a an aqueous slurry or suspension of calcium carbonate which is suitable for use in the toothpaste industry.

According to one embodiment of the present invention there is provided an aqueous suspension of particulate calcium carbonate for use in the manufacture of toothpaste compositions which includes, in addition to the calcium carbonate, one or more dispersing agents and/or one or more preservative agents and wherein the aqueous medium of the suspension has been treated so that the biological contamination of the suspension is not

greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA), for example standards set by Food and Chemical Codex and United States Pharmacopea.

All numerical values and ranges recited herein are to be understood as modified by the term "about," unless otherwise noted.

The suspension according to the invention may be a high solids suspension which is pumpable and flowable. The solids content of the suspension may be 50% or more, in many cases 60% or more, e.g. up to 80% or more, by weight. When measured by the Brookfield method using a Spindle 3 at 25°C, the viscosity may be 1000m Pa.s or less, e.g. 700mPa.s or less, especially from 100mPa.s to 500mPa.s.

All the raw materials used by the toothpaste industry must be food grade, that is, they must fulfill certain conditions demanded by various authorities which control the industry, especially the FDA. In particular, microbiological control levels of the raw materials must be compatible with those set for the pharmaceutical and food industries indicated by the FDA. The suspension according to the invention meets these requirements as they are presently expressed, and which are incorporated here for the first time of a calcium carbonate suitable for use in toothpaste industry in aqueous suspension form has several benefits. The problems described earlier are reduced or avoided. In addition, since the toothpaste manufacturing process requires large amounts of water for the addition of the other components of the toothpaste composition, and the calcium carbonate can be easily delivered from a storage tank to the manufacturing site by pumping, the aqueous suspension form is well suited to the toothpaste manufacturing process.

The calcium carbonate employed in the suspension according to the invention may be a PCC although it could alternatively be a product processed

from naturally occurring sources, generally by procedures involving grinding, i.e. a ground calcium carbonate or 'GCC'. The calcium carbonate may have an impurity content of not greater than 5%, preferably not greater than 3%, by weight. The calcium carbonate, if a PCC, may be prepared by any well known process. For example, PCC may be produced by carbonating an aqueous suspension of calcium hydroxide by addition of carbon dioxide.

The dispersing agent employed in the suspension according to one aspect of the invention may be any art recognized agent that is able to sufficiently disperse and reduce the viscosity of the suspension of calcium carbonate. In one embodiment, the dispersing agent comprises one or more inorganic or organic agents known in the art. Preferably, the dispersing agent comprises one or more sodium salts. The agent may, for example, comprise one or more of: a condensed phosphate salt, such as sodium hexametaphosphate; a silicate, e.g. sodium silicate; and mixtures thereof. The total amount of dispersing agent(s) employed may be from 0.1% to 6%, e.g. from 0.5% to 2%, by weight active based on the dry weight of calcium carbonate. We have found that a particularly suitable dispersing agent is a mixture of sodium silicate, 0.5% to 2.0%, preferably 0.8% to 1.2%, active by weight, and sodium hexametaphosphate, 0.1% to 0.7%, preferably 0.3% to 0.5%, active by weight, the weights being based on the dry weight of calcium carbonate. The dispersing agent may alternatively or in addition comprise any surface-modifying agent which will disperse the PCC in the slurry, such as surfactants or tensio-active agents.

The preservative agent employed in the suspension according to one aspect of the invention may be any substance known to inhibit microbiological growth. The agent to be used may be chosen from any preservatives known in the art. For example, the preservative agent may comprise one or more of: an

inorganic oxidizing preservative agent, such as sodium hypochlorite; a simple organic preservative agent, such as formaldehyde; or mixtures thereof. The total amount of preservative agent present in the suspension may be at least 0.005%, e.g. from 0.01% to 0.20%, especially from 0.01% to 0.10%, active based on the total suspension weight.

The calcium carbonate when in PCC form may be prepared in any well known manner, for example by carbonation of lime, e.g. by reaction of gaseous carbon dioxide with slaked lime in an aqueous suspension. The particles of the PCC may be in any form known in the art to be useful in toothpaste compositions. For example, the PCC particles may be in the rhombohedral or aragonite crystal form.

The aqueous suspension according to one aspect of the invention may be prepared as follows. A starting suspension of particulate calcium carbonate, e.g. a dilute PCC suspension formed following carbonation and neutralization having a solids content of not more than 40% by weight, e.g. from 10% to 30% by weight, is dewatered using any art recognized method. In one embodiment, a press filter or similar equipment may be employed. Preferably, a solids content of at least 50% by weight is obtained through dewatering. Alternatively, a cake may be produced. The particles of the cake are then dispersed by addition of a dispersing agent. Preferably, mechanical agitation, e.g. a high speed stirrer, is used to aid dispersion. The dispersion may be carried out in a suitable vessel such as a dispersion vessel, a blender or a dissolver.

The dispersing agent, which may comprise one or more suitable dispersant compounds, may be added in an amount sufficient to reduce the viscosity of the suspension to an appropriate value as defined earlier, suitable for its use in the toothpaste formulation. One or more preservative agents may

be added to the suspension to control or inhibit the development of microbiological contaminants in the suspension. A preservative agent such as sodium hypochlorite in the form of an aqueous solution is preferably employed as the preservative agent to be added to the suspension.

Water which has been treated by a process which inhibits microbiological growth may preferably be added to the suspension. The water may conveniently be that in which the dispersing agent or the preservative additive or preferably both is contained. Alternatively, the treatment of the water to inhibit microbiological growth may be performed after the addition of the water to the calcium carbonate, so that the entire calcium carbonate suspension is treated. The water may be treated by any known sterilization technique which will sufficiently reduce the microbiological activity level of the water. For example, the water may be treated using one or more of the following: known ultra high temperature treatments; sterilization; pasteurization; ozonation; or other processes known to reduce microbiological activity therein to an FDA accepted level. Preferably, the water is treated by ozonation.

The suspension product after addition of the dispersing agent and the preservative agent may be transferred to a storage tank. Where water contained in the suspension has been treated by ozonation, the suspension is left in the storage tank for a time sufficient to allow the amount of ozone remaining in the suspension to drop to acceptable levels. Preferably, the suspension is left for at least forty eight hours in the storage tank to allow active ozone to be eliminated to the fullest extent possible. Following such storage the suspension may be delivered to a toothpaste manufacturing plant by pumping (if the plant is local), by transport on a vehicle such as a slurry tanker, or by any other transport method.

The suspension may, after treatment as described above, optionally be further treated in a known manner to give a further concentrated product. This may be carried out through further dewatering or drying, e.g. using a vacuum or press cleaner and/or a dryer. Following such treatment, a solids content of up to 99.5% by weight may be obtained.

In another aspect of the present invention, there is provided a method of producing an aqueous suspension of calcium carbonate as described above wherein a dispersing agent and a preservative agent are added to a suspension of calcium carbonate and then water which has been treated by a process to reduce microbiological activity therein is added to the suspension.

In another embodiment, the dispersing agent and the preservative agent may be added to the suspension as a solution together with the treated water. Alternatively, the dispersing agent, preservative agent, untreated water, and calcium carbonate may be combined and then treated by one of the methods disclosed above.

Embodiments of the present invention will now be described by way of example only with reference to the following Example.

Example

50 tonnes of a (predominantly rhombohedral) PCC in an aqueous suspension having a 20% by weight solids content was prepared. The PCC had a minimum $CaCO_3$ content of 98.0%, i.e. not greater than 2% by weight of impurities, by weight and whiteness of 97%. The PCC suspension was filtered in a press filter, generating 14.285 tonnes of cake having a 70% solids concentration.

The cake was transported to a storage silo and afterwards transferred ready for addition to a dispersion vessel having a capacity of 2000 litres and

fitted with a stirrer operating at a speed of 1500 rpm. 236 kg of an aqueous solution which had been pre-treated as described below was added to and mixed with 3,064.26 kg of the PCC cake in the vessel. The solution added to the vessel contained as additives (i) sodium silicate in an active amount of 1.0% based on the dry weight of PCC present; (ii) sodium hexametaphosphate in active amount of 0.4% by weight based on the dry weight of the PCC present and (iii) sodium hypochlorite preservative agent in an active amount of 0.015% by weight based on the total suspension weight. The base water employed to provide the solution containing the additives (i), (ii) and (iii) was previously ozonated for one hour, to achieve a solution meeting current international microbiological control standards. This pre-ozonated solution still contained active ozone when it was added to and mixed with the PCC cake.

The contents of the dispersion vessel were stirred for about one hour to ensure complete dispersion of the PCC. After that period, the obtained suspension had a viscosity of 250 mPa.s (measured by the well known Brookfield method using a Spindle 3, at 25°C). This suspension was kept in storage tanks for a period of forty-eight hours until there had been complete elimination of the active ozone from the suspension before delivery of the suspension to a toothpaste production plant.

CLAIMS

1. An aqueous suspension of particulate calcium carbonate for use in the manufacture of toothpaste compositions wherein the biological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA).

2. An aqueous suspension of particulate calcium carbonate for use in the manufacture of toothpaste compositions wherein the aqueous medium has been treated so that the biological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA).

3. An aqueous suspension according to claims 1 or 2 wherein the suspension further comprises one or more dispersing agents.

4. An aqueous suspension according to claims 1 or 2 wherein the suspension further comprises one or more preservative agents.

5. An aqueous suspension for use in the manufacture of toothpaste compositions comprising:

particulate calcium carbonate;

one or more dispersing agents; and

one or more preservative agents;

wherein the aqueous medium has been treated so that the biological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA).

6. A suspension according to any of the preceding claims wherein the calcium carbonate comprises a precipitated and/or ground calcium carbonate.

7. A suspension according to any of the preceding claims wherein the calcium carbonate has an impurity content not greater than 5% by weight.

8. A suspension according to claim 7 wherein the calcium carbonate has an impurity content not greater than 3% by weight.

9. A suspension according to any one of the preceding claims wherein the solids content of the suspension is from 50% to 99.5% by weight.

10. A suspension according to claim 9 wherein the solids content of the suspension is from 80% to 99.5% by weight.

11. A suspension according to any one of the preceding claims wherein the suspension is flowable and/or pumpable.

12. A suspension according to any one of the preceding claims wherein the suspension has a viscosity of not greater than 1000mPa.s.

13. A suspension according to claim 12 wherein the suspension has a viscosity of from 100mPa.s to 500mPa.s.

14. A suspension according to any of claims 3 or 5 to 13 wherein the dispersing agent comprises one or more sodium salts.

15. A suspension according to any of claims 3 or 5 to 13 wherein the dispersing agent comprises a condensed phosphate and/or a silicate.

16. A suspension according to claim 15 wherein the dispersing agent comprises from 0.1% to 0.7% active by weight of sodium hexametaphosphate and from 0.5% to 2.0% by weight of sodium silicate, the weights being based on the dry weight of calcium carbonate present.

17. A suspension according to claim 16 wherein the dispersing agent comprises from 0.3% to 0.5% active by weight of sodium hexametaphosphate and from 0.8% to 1.2% active by weight of sodium silicate, the weights being based on the dry weight of calcium carbonate present.

18. A suspension according to any of claims 4 to 17 wherein the preservative agent comprises an oxidizing inorganic chemical or a simple organic chemical.

19. A suspension according to claim 18 wherein the preservative agent comprises sodium hypochlorite or formaldehyde.

20. A suspension according to claim 18 or claim 19 wherein the amount of preservative agent present is from 0.005% to 0.2% by weight based on the total suspension weight.

21. A suspension according to claim 20 wherein the amount of preservative agent present is from 0.01% to 0.1% by weight based on the total weight of the suspension.

22. A suspension according to any one of the preceding claims wherein the aqueous medium of the suspension has been treated by a process to reduce microbiological activity therein.

23. A suspension according to claim 22 wherein the treatment process comprises a sterilization, pasteurization or ozonation process, or a combination thereof.

24. A suspension according to claim 23 wherein the treatment process comprises ozonation.

25. A method of producing an aqueous suspension of calcium carbonate for use in the manufacture of toothpaste compositions comprising:

adding a dispersing agent and a preservative agent to a suspension of calcium carbonate; and

adding to the suspension water that has been treated by a process to reduce microbiological activity therein.

26. A method of producing an aqueous suspension of calcium carbonate for use in the manufacture of toothpaste compositions comprising:

providing an aqueous solution of water, a dispersing agent and a preservative agent; and

adding to a suspension of calcium carbonate the aqueous solution of water;

wherein the water has been treated by a process to reduce microbiological activity therein.

27. A method according to claims 25 or 26 wherein the suspension prior to addition of the dispersing agent and the preservative agent is dewatered to have a solids content of at least 50% by weight.

28. A method according to claim 27 wherein the suspension is dewatered to produce a cake.

29. A method of producing an aqueous suspension of calcium carbonate for use in the manufacture of toothpaste compositions comprising:

providing an aqueous suspension of calcium carbonate containing a dispersing agent and a preservative agent; and

treating the aqueous suspension by a process to reduce the microbiological activity therein.

30. A method according to claims 25 to 29 wherein the microbiological activity such that the microbiological contamination of the suspension is not greater than the maximum allowable levels indicated by the US Food and Drugs Administration (FDA).

31. A method according to claims 25 to 30 wherein the treatment process comprises a sterilization, pasteurization or ozonation process, or a combination thereof.

32. A method according to claim 31 wherein the treatment process comprises an ozonation process.

33. A method according to any one of claims 25 to 32 wherein the suspension is dewatered following addition of the dispersing agent and the preservative agent.